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Study of the Simultaneous Elimination of Phosphates and Heavy Metals Contained In Dairy Wastewater by A Physical-Chemical and Biological Mixed Process; Consequences on the Biodegradability.

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Abstract

The study examines the influence of the presence of heavy metals presence on the elimination of phosphates in dairy wastewater. It focuses on the phosphates precipitation in a stoichiometric dose of ferric chloride and is made of two main parts:

- Without contribution of heavy metals.
- With contribution of heavy metals (lead and cadmium).

In both parts, the removal was carried out at first in a physical-chemical way by the precipitation of phosphates with ferric chloride; the residual phosphates concentrations were then biologically eliminated by activated sludge taken from an urban wastewater treatment plant.

For the tests conducted with heavy metals presence, lead and cadmium concentrations between (10 and 100 mg / L) were added to the wastewater kept for our study. The main results show:

- Good phosphate precipitation with the use of ferric chloride.
- The combination of biological and physical-chemical processes is possible since a removal of 69% in presence of Lead (Pb) and 93% in presence of Cadmium (Cd) were obtained.
- Maximum removals of 100% lead (for 100 mg / L of Pb) and 94% cadmium (for 50 mg / L of Cd) were obtained after phosphates precipitation by the ferric chloride.

Moreover, in an attempt to assess the performance of the biological treatment by activated sludge combined to the physical-chemical process on the removal of heavy metals in the water to be treated. The evolution of the Cadmium's concentration according to time was followed in both supernatant and sludge. This was made possible by a kinetic study in batch. In this case, the results show:

- Cadmium removal from about 81% in the supernatant.
- Good retention of cadmium in the sludge.

The synthesis of all the results obtained during this study shows that the combination of physical-chemical and biological processes improves the removal of phosphates in the absence and presence of heavy metals.

Keywords: physical-chemical, biological, phosphorus removal, precipitation of phosphates, heavy metals.

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1. Introduction

Water, which is essential for life and any economic activity, is used for domestic, industrial and agricultural purposes. Following these different uses, water is often polluted. Its release into the environment as more or less polluted effluents may, in the short or the long term, lead to serious nuisance on public health and hygiene and on the environment as well. It may have a significant economic impact. It is therefore important to treat this wastewater. Increasing

water pollution due to a worldwide growing population requires the use of various liquid waste management schemes to ensure the survival of water bodies across the globe. Among the pollutions, the nitrogen and the phosphorus can stimulate the development of certain plants and contribute to the eutrophication phenomenon [1]. The reduction of phosphorus flux brought into aquatic environments by different discharges is an asset to fight against the eutrophication that disturbs the balance and the use of the ecosystems.

When we consider the frequent droughts, we have a better understanding of the dramatic character of the problem of water resources.

The question of the wastewater treatment took on an increasing importance at the beginning of the seventies, considering the general concern expressed all around the world about the growing problem of the pollution of the human environment, the atmosphere, the rivers, the lakes, the oceans, and groundwater by the urban, agricultural, and industrial wastes.

Industry contributes for a very important part in water pollution by rejecting toxic metals and chemical products. The concentration of the environment by heavy metals is one of the most serious facts of our civilization. Indeed, the majority of heavy metals found in water are considered as poisons which mean substances that cause damage or disruption of body functions, leading to harmful effects. Man can be a direct or indirect victim if such products reach the ground water supply.

The dairy industry is considered as one of the polluting industries because dairy effluents may constitute a risk of pollution when they are discharged without proper treatment in the surface water or groundwater. Indeed, they contain nitrogen and phosphorus concentration that exceeds discharge standards and is up to ten times higher than the effluents of home septic tank. When they are released into the environment, these products pollute groundwater and streams. They often cause a growth of algae and bacteria that consume oxygen and water and eventually suffocate the receiving environment, which leads to the gradual disappearance of fishes. When these discharges percolate into groundwater, they alter the drinking water quality by increasing for example, the content of nitrates and phosphates. To avoid these problems, we must eliminate the pollution of the water before its release into the environment [2, 3].

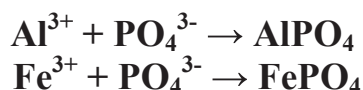
Dairy processing wastewater has unique properties which differentiate it from domestic wastewaters. It has high levels of COD and dissolved reactive phosphorus and nitrogen. Algiers dairy industry wastewaters can have phosphorus contents up to 130 mg/L, on the average 90% of total phosphorus was in form of orthophosphates (PO_4^{3-}), probably

originating from the high levels of phosphoric acid used for cleaning the process equipment.

The aim of this study was to examine the influence of the presence of heavy metals (especially lead and cadmium) on the elimination of phosphates contained in a dairy wastewater.

Phosphorus removal from wastewater can be realized according to physical-chemical or biological means [4, 5].

To this day, the most common technique of phosphate removal is chemical precipitation with iron or aluminum salts [6-8](Wang et al., 2005, Xie et al. 2004, Katz et al., 2008). As shown in the following chemical reactions:



The physical-chemical precipitation is simple to implement, reliable and efficient but presents several disadvantages: increased operating costs due to the consumption of chemical reagents and the corrosiveness of the product; excess sludge production, salts rejection in the receiving environment [9-11]. The biological excess elimination of phosphorus, commonly called biological phosphorus removal is based on the alternation of anaerobic / aerobic phases and leads to a phosphate accumulation by activated sludge biomass beyond the growth metabolic requirements [12, 13].

While an activated sludge normally contains 1.5 to 2% phosphorus reduced to dry material, phosphate removal sludge may contains 3.5 and even 10% of phosphorus. In addition to its ecological asset, the biological phosphorus removal presents many advantages compared to chemical phosphorus removal, especially in the generation of a sludge quantity that is similar to a conventional process, agricultural valuation of sludge enriched with directly available phosphorus, reduction of the cost bound to chemical reagents consumption, unimportant additional cost for some processes.

In this work, we have tried to study the influence of the heavy metals presence on the phosphates removal in dairy waste water. The study was realized in two main parts:

-Without contribution of heavy metals.

-With contribution of heavy metals.

In both parts, the removal is carried out in two stages:

* In a physical-chemical way, by a ferric chloride precipitation.

* The residual phosphates are then eliminated in a biological way by activated sludge taken from a sewage plant aeration tank.

2. Materials and Methods

Experiments realized in this study include two sets of tests:

- The phosphates removal is achieved by ferric chloride (FeCl_3 , $6\text{H}_2\text{O}$) precipitation at a stoichiometric dose.
- After the results, we pursue our work by testing the supernatant on one hand and the centrifuged sludge on another hand.

*** On the supernatant**

We proceed to the biodegradability tests by biological phosphate elimination kinetics according to different heavy metals concentrations.

*** On the centrifuged sludge**

We proceed to a successive dilution tests in order to study the phosphates release.

2.1. Experimental device

The experiments were performed using a laboratory flocculator (Jar-test), its simplicity of use and good results reproducibility make it a well suited device to the precipitation study. This jar-test includes 06 agitators whose rotation speed can vary between 10 and 200 r/min. The blades are from helixes type. The beakers volume is 1L.

A preliminary study [14] allowed us to obtain the optimum operating conditions as follows:

- A rapid stirring phase of 2 minutes during which the coagulant (ferric chloride) and the water to treat are strongly shaken to distribute the reagent.
- A slow stirring phase of 10 min at a speed of 60 r/min.
- A settling phase of 10 min.

2.2. Analytical methods

The analytical methods used in this work are AFNOR standards based [15]. Each collected sample was analyzed for COD, orthophosphates, according to a colorimetry method by *HACH DR 2010* spectrophotometer.

The heavy metals were dosed through atomic absorption spectrophotometer. The solutions that were taken were filtered and diluted then acidified with few nitric acid (HNO_3) drops to be dosed. The metal concentrations are determined by a flame atomic absorption spectrophotometer with a wave length of 217.2 nm for the lead and 628.8 nm for the cadmium.

Average composition of the wastewater to treat:

The effluent to treat is a dairy wastewater whose composition is reported in table 1.

Table 1: Average composition of wastewater

DCO (mg/L)	769-1000
DBO ₅ (mg/L)	380-560
N-NH ₄ ⁺ (mg/L)	0.89-28
N-NO ₃ ⁻ (mg/L)	0.7-1.5
P-PO ₄ ³⁻ (mg/L)	5.6-56
pH	4.2-8.3
K (DCO/DBO ₅)	1.78-2.02

3. Results and Discussion

3.1 - phosphates precipitation without heavy metal contribution

After proceeding to the phosphates precipitation by the ferric chloride, and in order to proceed to the biodegradability tests on the supernatant, the elimination kinetics of residual phosphates with two different volumes of sludge were performed, in order to determine the volume of sludge allowing us to have an optimal global elimination (physical-chemical and biological).

As such, two volumes of sludge were tested: 250 and 500 mL.

After adjustment of pH value to 7, by adding an acid (HCl, 0.1N) or base (NaOH, 0.1N), we fill each beaker with 1 L of water to treat and add the necessary ferric chloride quantity for the phosphates precipitation in the stoichiometric dose. The beakers are subjected to a rapid stirring for 2 min at a speed of 200 r / min. This step is followed by a slow stirring at a speed of 60 r / min for 10 min. Stirring is then stopped and the settling phase is then affected, it lasts 10 min. At the end of the settling phase, the supernatant is separated from the deposit in each of the beakers.

3.1.1. Tests on the supernatant:

A: Kinetics realized with 250 mL of sludge

A volume of 750 mL of supernatant is mixed with 250 ml of activated sludge (taken from the aeration tank of Staoueli wastewater treatment plant) in a beaker of 1L which is subjected to a permanent stirring. Samples taken every 10 min for 1 hour, allow us to follow the residual phosphate concentration evolution over time. The results obtained are given in table 2;

pH=7, T= 25°C

$[\text{PO}_4^{3-}]_{\text{rw}} = 28.4 \text{ mg/L}$; rw= raw water

$[\text{PO}_4^{3-}]_{\text{ap}} = 21.2 \text{ mg/L}$; ap= after precipitation by Fe Cl_3

We note a phosphate removal in the range of 25.35%, after precipitation. Indeed, from 28.4 mg / L in the raw water, the phosphate concentration is crossed to 21.2 mg/ L.

Table2: Phosphates evolution over time for 250 mL of sludge

Time (min)	Residual Phosphates concentration (mg/L)	Phosphates Removal (%)
0	21.2	0
10	20	5.66
20	19.2	9.43
30	19	10.37
40	18.6	12.26
50	18.4	13.20
60	17.8	16.04

The phosphates biologic elimination efficiency obtained is 16.04%. The global elimination obtained in this case is then 41.4%.

B: Kinetics performed with 500 mL of sludge

In this case, we mix 500mL of sludge and 500 mL of supernatant and proceed to the same kinetics. The operating conditions for these tests are:

pH=7, T= 25°C

$[\text{PO}_4^{3-}]_{\text{rw}} = 56.8 \text{ mg/L}$; rw= raw water

$[\text{PO}_4^{3-}]_{\text{ap}} = 47.2 \text{ mg/L}$; ap= after precipitation by Fe Cl_3

We note a phosphate removal in the range of 25.35%, after precipitation. Indeed, from 56.8 mg / L in the raw water, the phosphate concentration is crossed to 47.2 mg / L. The obtained results are given in Table 3.

Table 3: Phosphates evolution over time for 500 mL of sludge

Time (min)	Residual Phosphates concentration (mg/L)	Phosphates Removal (%)
0	47.2	0
10	44	6.78
20	43.6	7.63
30	42.8	9.32
40	42.6	9.74
50	42.4	10.17
60	40	11.02

The biological phosphates removal efficiency obtained in this case is 11.02%. The global elimination obtained is 27.92%.

All obtained results are reported in figure 1 that gives the phosphate removal efficiency evolution according to the time for the two sludge volumes tested.

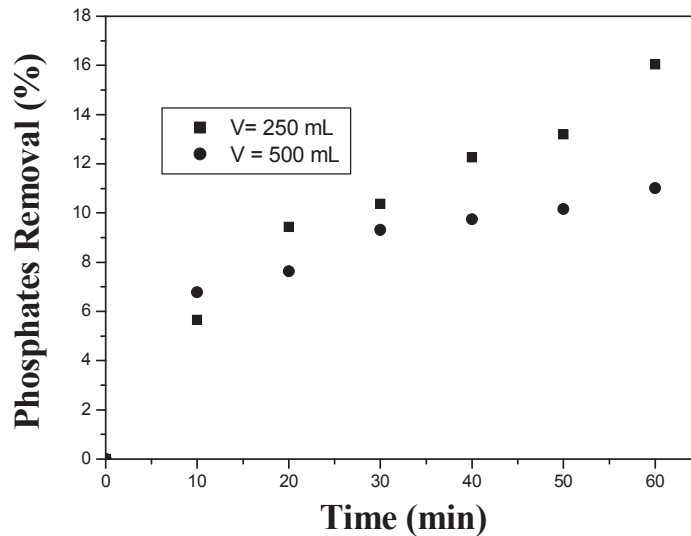


Figure 1. Evolution of phosphate removal efficiency according to the time for the two sludge volumes tested.

Examination of this figure shows that the biological removal obtained with 250 mL of sludge (16.04%) is more important than that obtained with 500 mL of sludge (11.02%). Since the best performance of phosphorus removal was obtained with a sludge volume of 250 mL, the adopted operating conditions for the rest of our tests are:

- A sampling time of 60 min;
- A sludge volume of 250 ml

3.1.2. Tests on centrifuged sludge:

After removing the supernatant in each beaker, the deposit (sludge) is removed and spun in turn. We then proceed to successive dilutions with different distilled water volume to study the presence of phosphate in the sludge.

In Table 4 are recorded the obtained results.

Table 4. Evolution of phosphate concentration in the sludge according to dilution volume.

Dilution volume (mL)	10	20	40	60	80
PO_4^{3-} Concentration (mg/L)	10.2	8	7	6.8	5.6

These results show that a volume of dilution of 10 mL allows to obtain the best phosphates concentration (10,2 mg/L), this allows us to assert that the sludge can trap phosphates on one hand, and the complementarity of the physical-chemical and biological processes on the other hand.

3.2. Phosphates precipitation with heavy metals contribution

The previous tests allowed us to observe a good phosphates precipitation by FeCl_3 and a good biological removal with an activated sludge volume of 250 mL.

In order to study the heavy metals influence on phosphate removal, we doped water with a metal (lead) and followed the phosphate removal efficiency according to different concentrations of this element. All tests are realized following the same experimental protocol adopted at the previous trials. After phosphates precipitation in the stoichiometric dose, we take 750 mL of supernatant that we mix with 250 ml of activated sludge, and then add the metal concentration that we want to study.

3.2.1 Phosphates removal kinetics in lead presence

Lead (Pb) concentrations studied are; 10, 30, 50, 70 and 100 mg/L. These concentrations are well above the discharge standards of this element in water which is: 0.5 mg / L for Pb according to the executive decree n ° 93-160 of 14-07-1993 of the Algerian Republic Official Journal.

The principal obtained results are recorded in table 5 and figure 2.

Table 5. Evolution of biological phosphates removal according to time for different Pb concentrations.

Time (min)	10 mg/L Pb		30 mg/L Pb		50 mg/L Pb	
	PO ₄ ³⁻ (mg/L)	% Elim(%))	PO ₄ ³⁻ (mg/L)	% Elim(%))	PO ₄ ³⁻ (mg/L)	% Elim(%))
0	22	0	19.2	0	30.4	0
10	21.2	3.63	18.8	2.08	23.2	23.68
20	19.2	12.72	17.6	8.33	21.6	28.94
30	19	13.63	17.6	8.33	20.8	31.57
40	18.8	14.54	16.4	14.58	19.2	36.84
50	18.6	15.45	15.4	19.79	-	-
60	18	18.18	14.6	23.96	12.8	57.89

For lead concentrations of 70 and 100 mg/L, the phosphates concentrations after precipitation are so low (below the discharge standard, a concentration below 1 mg/ L) that we are unable to undertake the phosphates removal kinetics. The values of the table 5 show that the elimination is all the more important as the lead concentration is important. So average eliminations of 11.16, 13.69 and 29.82 % were respectively obtained for 10, 30 and 50 mg / L from lead concentrations.

We think that this elimination is due to "a cationic substitution" so to an ions exchange between lead and iron of ferric chloride so that the lead is removed by precipitation in the form of Pb₃ (PO₄)₂ (Pb phosphate). More deepen analysis could certainly bring more specifications.

Such observation is made compared to a study performed by [16] about Pb immobilization tests by hydroxyapatite which is a calcium phosphate which chemical formula is Ca₅(PO₄)₃(OH), commonly known as PAH.

The tests were undertaken following two contradictory theories:

* According to the first theory, the reaction mechanisms would be dominated by the HAP dissolution followed by precipitation.

* According to the second theory, the lead (Pb) immobilization would be done through an ion exchange (or cationic substitution) between the ions Ca⁺² and Pb⁺² followed by precipitation. In this case, the Pb totality is immobilized (complete retention) when the pH is between 7 and 7.4. For our part, we look to the second theory because our tests pH is 7.

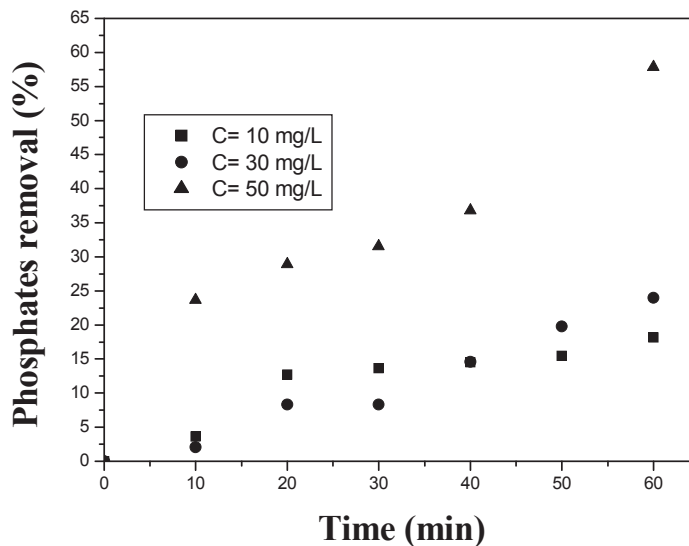


Figure 2 .Evolution of biological phosphates removal according to time for different Pb concentrations.

3.2.2. Consequences of the lead presence on the organic matter biodegradation

For an effective phosphorus removal, it is imperative that the wastewater contains a sufficiently high organic load, mainly in rapidly biodegradable COD, because this organic matter fraction constitute the biomass phosphorus removal substrate and phosphorus removal depends on this biomass development [17].

So the biologic phosphates removal from water is done at the same time as the organic matter degradation. However, some molecules can inhibit the microorganism's metabolic functions or be difficult to degrade because of their complexity. Moreover, other substances or compounds such as heavy metals can interfere with biological processes and act as poisons.

The biological phosphorus removal kinetics in presence of Pb showed good

phosphate removal in presence of different concentrations of this element.

Therefore, we proposed to evaluate the presence of Pb effect on the organic matter degradation by following the COD evolution according to time for a Pb concentration of 50 mg / L.

The experimental protocol is similar to the one in the previous tests. After phosphates precipitation in a stoichiometric dose, we take 750 mL of supernatant that we mix with 250 ml of activated sludge, and then add 50 mg / L of Pb. The mixture is stirred. Samples taken according to time, allowed us to follow the COD evolution. The obtained results are given in table 6 and figure 3.

Table 6: Evolution of COD removal according to time in presence of 50 mg/L of lead.

t (min)	DCO (mg/L)	% Elimination (%)
0	769	0
10	700	8.97
20	650	15.47
30	580	24.58
40	475	38.23
50	340	55.79
60	202	73.73

These results show a decrease in COD values according to time in Pb presence., Indeed, 769 mg/L at $t = 0$, COD passes to 202 mg/L at $t = 60$ min which gives a performance elimination in the range of 73.73%. This confirms the fact that the phosphates removal is made at the same time as the organic matter degradation even in Pb presence. In other words, lead's presence doesn't inhibit the organic matter degradation.

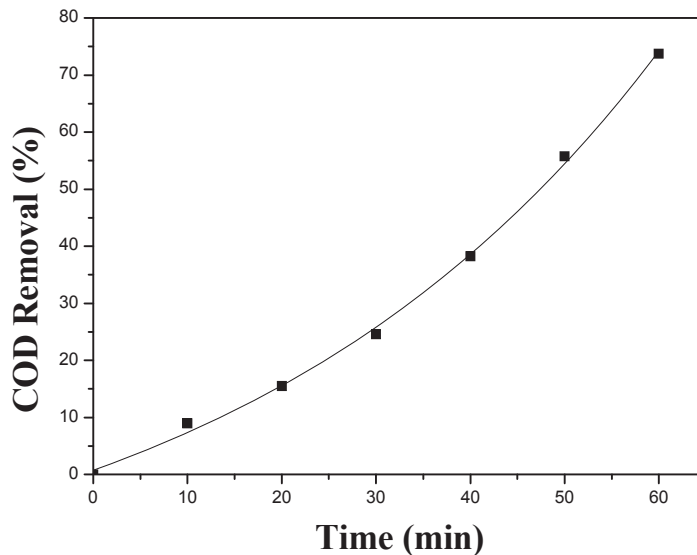


Figure 3. COD removal efficiency evolution according to time in the lead (Pb) presence.

Conclusion

During this work, we were interested in the study of the influence of heavy metals presence on the elimination of phosphates contained in a dairy wastewater.

The main obtained results show:

- A good phosphate precipitation with the ferric chloride use.
- An additional biological removal to the physical-chemical elimination because 25.35% of phosphates are eliminated by the physical-chemical process, and 16.04% are with the biological process leading to an overall removal of 41.4%.
- Phosphates trapping by activated sludge.
- Phosphates removal even in presence of significant lead (Pb) concentrations.
- The elimination is as much important as the Pb concentration is important. It's thus for a same reaction time of 60mn; 14.6; 23.96 and 57.89% of phosphates are removed for 10, 30 and 50 mg /L Pb, respectively.
- An organic matter degradation expressed in COD at the same time as the phosphates biological removal even with Pb presence in the water. In other

words, Pb presence doesn't inhibit the organic matter degradation and phosphates assimilation. We also think that deeper researches on the mechanisms of physical-chemical phosphates removal in the heavy metals presence are necessary.

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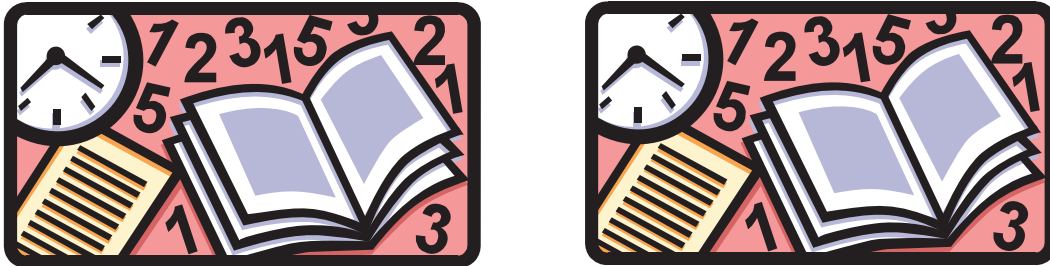


Fig. 1. (a) first picture; (b) second picture

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Acknowledgements

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References

- [1] Van der Geer J, Hanraads JAJ, Lupton RA. The art of writing a scientific article. *J Sci Commun* 2000;**163**:51–9.
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Appendix A. An example appendix

Authors including an appendix section should do so after References section. Multiple appendices should all have headings in the style used above. They will automatically be ordered A, B, C etc.

A.1. Example of a sub-heading within an appendix

There is also the option to include a subheading within the Appendix if you wish.